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# A Materials-Science Based Approach to Phenol Emissions from a Flooring Material in an Office Building

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## ABSTRACT

After several years of indoor air quality complaints in an office building, it was hypothesized that phenol emissions from an epoxy floor-leveling material were the source of the complaints. A materials-science based study was performed to ascertain whether phenol, or any other volatile organic compound, was being emitted from the floor-leveling material. The chemical composition and physical properties of the leveling material were determined using a variety of analytical procedures typical of a materials-science based approach. It was concluded that the floor-leveling material contained phenol, and that the measured concentrations ranged from 0.25% to 0.52% of the material's mass. Several strategies for mitigating the phenol emissions were considered, and it was concluded that the most practical strategy would be to remove the floor-leveling material from the building and replace it with a portland cement-based material.

## INTRODUCTION

The diagnosis of indoor air quality complaints employs a variety of tools, including building walk-throughs, system analyses and inspections, and air sampling (1-4). In many cases, these approaches do not uncover the causes of the complaints, and more detailed study is required. In particular, contaminant sources in buildings that are associated with interactions of materials can be quite difficult to sort out using traditional diagnostic procedures. A number of case studies have been reported where building materials, in particular concrete floor slabs and floor coverings, have been suspected of interacting (5-7). This paper reports on a situation in an office building in which an epoxy-based floor leveling compound emitted phenol into the indoor air, leading to a large number of indoor air quality complaints (8). After many indoor air quality investigations, phenol concentrations were measured in the building and the floor-leveling material was identified as a suspected phenol emission source. An independent assessment of the floor-leveling material was requested by various organizations involved with the building, and a materials-science based approach was employed to assess whether the floor-leveling material was a likely source of the phenol emissions, and, if so, to recommend remedial strategies.

## BUILDING HISTORY

The SSMC-1 Building is located in Silver Spring, MD and houses employees of the National Oceanic and Atmospheric Administration (NOAA). Base-building construction began in 1984, and was completed in 1986. The floor below the lobby, a portion of the fifth floor and all of Floors 6 through 9 were occupied in 1988. The remainder of the building (Floors 2 through 4 and the balance of Floor 5) was scheduled to be occupied in June 1989; however, occupancy was not completed until January 1990 due to the following events.

In March 1989, while furniture was being installed on Floor 4, significant deflections were observed in the floor slab. The furnishing was delayed until the structural implications of the deflections could be ascertained. This delay was changed into a full halt when excessive deflections were noted on all the floors. A full-scale structural survey of the building was then undertaken. As a result of this study, the concrete floor slabs on Floors 2 through 9 were reinforced with steel brackets in areas considered to be structurally deficient. The survey team also found that the builder had applied large amounts of an inorganic floor-leveling material on all nine floors. This floor leveling material was removed from Floors 2 through 4 and most of Floor 5 during the structural retrofit of the building and was replaced by a floor-leveling material containing an epoxy binder (hereinafter called the leveling material).

Soon after Floors 2 through 5 were occupied in January, 1990, building occupants began to complain about eye, nose and throat irritation, nausea and fatigue. In February 1990, soon after receiving the initial complaints, NOAA's management commissioned an indoor air quality survey of SSMC-1. Since this survey, at least five more indoor air quality surveys were performed. The early surveys monitored comfort parameters (air movement, relative humidity, and temperature), carbon dioxide, carbon monoxide, and a few other air quality parameters. The results from these surveys served to establish baseline data, but did not identify any parameter likely to be causing the complaints. Instead, they emphasized the need for changes related to thermal comfort and, in response, improvements were made in the HVAC system. The surveys did provide indirect evidence, however, that the source of the indoor air quality complaints could be a volatile organic compound. One survey reported "very strong chemical-like odors" on Floors 2 through 5; another mentioned "formaldehyde-like odors"; and a third noted "a new plastic or vinyl odor vapor" on Floors 2, 3, 4, and 7 (8).

Since the improvements in the HVAC system did not lead to a cessation of the indoor air quality complaints, another survey was commissioned which included analyses for phenol. This survey measured concentrations of phenol in the air of about 40 $\mu g/m^3$ to 80 $\mu g/m^3$ on floors 3, 4 and 5, and less than 20 $\mu g/m^3$ on floors 7 and 8. It was concluded that "the distinct chemical solvent type odor noticeable on the 3rd and 4th floors is associated with phenol, which is a component of the floor-leveler used on these floors" (9,10). These reports also presented results of a headspace analysis of the flooring material (carpet, adhesive, and leveling material), which also indicated the presence of phenol. Thus, these latter surveys provided evidence of a connection between phenol in the air and phenol emissions from the leveling material. Further, since the phenol concentrations in the air were highest on Floors 3 through 5, which had the most indoor air quality complaints, there was strong evidence that phenol emissions were associated with the complaints. In August of 1993, the General Services Administration (GSA), who owned and operated the building, and NOAA requested that the National Institute of Standards and Technology (NIST) investigate whether phenol, or any other volatile organic compound, was being emitted by the epoxy floor-leveling material and, if so, to recommend remedial strategies for eliminating or mitigating the emissions.

## EPOXY FLOOR-LEVELING MATERIAL

The epoxy floor-leveling material is made by blending four to ten parts of sand with one part of an epoxy binder. The epoxy binder is a two-part system which is made by mixing four parts epoxy resin with one part hardener. The hardener contains phenol (1 to 5% by volume) and a number of amine crosslinking agents which convert the normally linear epoxy resin into a three-dimensional, crosslinked network. Phenol controls the size of the crosslinked network by terminating the polymerization reaction, which in turn controls the flexibility of the leveling material. Phenol molecules taking part in the polymerization reactions become strongly bound to the epoxy and cannot easily be emitted into the atmosphere. However, chemical reactions are seldom 100% efficient and, hence, some portion of the phenol molecules do not take part in the

chemical reaction. Unreacted phenol molecules are not strongly bound and are likely to diffuse out of the leveling material. According to the manufacturer, the maximum amount of phenol present in a properly mixed leveling material in which ten parts of sand are mixed with one part of binder is approximately 0.02% by mass. The amount of unreacted or free phenol in a properly mixed and cured leveling material (that is, phenol which has not taken part in the polymerization reaction), therefore, should be much less than 0.02% by mass.

## SAMPLING PLAN

A sampling plan was designed to determine the phenol content of the leveling material and to detect large differences in the phenol content between floors. The plan involved drilling cores of leveling material on Floors 2 through 5, and was devised so that all areas containing the leveling material had an equal opportunity of being sampled. Details of the sampling plan are contained in reference (8). After the cores were brought back to the laboratory, each core was crushed in a mortar and pestle until the maximum particle size was less than 3 mm in diameter. The particles were then mixed and a one-gram sample, a small portion of the total mass of the specimens, was removed for analysis by GC/MS (gas chromatography/mass spectrometry). The remainder of the material was ground into a powder in a ball mill.

For each floor, the twelve powdered specimens were randomly assigned to one of two groups, and, within each group of six specimens, a composite specimen was prepared by mixing an amount of material from each specimen that was proportional to its depth. This was done to ensure that the contribution from each core was approximately proportional to the amount of material it represented. Thus, two composite specimens were made for each floor.

## RESULTS FROM STUDIES OF LEVELING MATERIAL

Thermogravimetric analysis (TGA) was used to determine the mass fractions of sand, volatile organic compounds, and epoxy binder in the cured leveling material. The procedure entailed placing 45 mg of the powdered composite material onto a thermobalance and continuously monitoring the mass loss of the material, while heating it from room temperature to 600 °C in nitrogen, and from 600 °C to 800 °C in air. TGA curves, plots of percent mass versus temperature, were generated for each of the composite samples. The following discussion refers to the results obtained for one of the samples from Floor 4.

In the presence of air and at temperatures of 800 °C and above, all of the organic materials in the powdered leveling material are decomposed and volatilized, leaving only inorganic materials. For the composite sample from Floor 4, a mass fraction of approximately 93.5% of the leveling material was non-volatile inorganic materials most likely sand. At temperatures between 200 °C and 800 °C, the epoxy binder undergoes thermal degradation causing the epoxy resin to break down into small molecular units by pyrolysis or oxidation, which are then volatilized along with the high boiling organic compounds, like amines. Based on the TGA curves, the epoxy binder comprised a mass fraction of approximately 5.7% ± 0.6% of the leveling material. The remaining 0.8% mass fraction of the volatile material (100% - 93.5% - 5.7%) was volatilized between room temperature and 200 °C. This would include volatile organic compounds like phenol. Of this 0.8% mass fraction, about 0.2% was determined to be water. Therefore volatile organic compounds constituted about 0.6% ± 0.1% of the mass of the fourth floor sample. Samples from the other floors ranged from about 0.5% to 0.8% with an uncertainty of 0.1%. Reference (8) contains data for all the samples.

The volatile organic compounds emitted between room temperature and 200 °C were identified using both GC/MS and LC/MS (liquid chromatography/mass spectrometry) techniques. From the GC/MS analysis, it was concluded that the predominant volatile organic compound

liberated from the leveling material between room temperature and 200°C was phenol. The GC/MS was not equipped to detect amines, so a complementary LC/MS experiment was performed which could detect the presence of both amines and phenol. The dominant volatile organic compound in the LC/MS tests was determined to be phenol. In addition, approximately 5% of the mass of the condensate was triethanolamine.

The GC/MS and LC/MS procedures were designed to determine qualitatively the volatile organic emissions from the leveling material. Estimates of the phenol concentration in the leveling material were obtained from Soxhlet extractions at 90 °C in water and 25 °C in methylene chloride. The concentration of phenol in the extracts was quantified by ultraviolet absorption. The results from the Soxhlet extractions yielded concentrations of phenol in the composite specimens taken from Floors 2 through 5 ranging from 0.25% to 0.52% ± 0.01% of the mass of the leveling material. As noted earlier, the maximum amount of phenol in the leveling material, when properly mixed and cured, is 0.02% by mass.

An important question was whether the phenol could diffuse out of the leveling material or whether it was strongly bound in the epoxy matrix and not able to escape. The low temperatures of the Soxhlet extractions strongly suggested that most of the phenol was "loosely-held" and, given enough time, would diffuse out of the leveling material and into the atmosphere. This suggestion was reinforced by the results from another experiment in which the emissions from the granulated leveling material were measured by GC/MS immediately after the material was removed from its container and again after purging the same specimen with nitrogen for one hour. Over this one-hour period, the concentration of phenol in the emissions decreased by a factor of five indicating that most of the phenol was not strongly bound and would eventually diffuse out of the leveling material. It was concluded from these experiments that most the phenol molecules are "loosely held" within the matrix of the leveling material.

Concern was expressed that, since the leveling material was in intimate contact with the concrete floor slabs, the concrete might be contaminated with phenol and be a source of phenol emissions if the leveling material was removed. To test this possibility, cores of the concrete beneath the leveling material were taken from Floor 4. The leveling material was separated from the concrete. Starting from the top of the core, three 4-mm thick disks were cut from the concrete cores using a diamond saw. The wafers were crushed into coarse particles for analysis by GC/MS. The GC/MS results showed no detectable amounts of phenol in any of the cores. This was not unexpected since portland cement concrete contains calcium hydroxide, a strong base, which combines with phenol, a weak acid, to form a salt, calcium phenolate.

## POSSIBLE REMEDIAL STRATEGIES

From the material analyses, it was concluded that significant concentrations of phenol were present in the leveling material on Floors 2 through 5, and that phenol was emitted, and would continue to be emitted, into the atmosphere from the leveling material. Four remedial strategies were considered for mitigating or eliminating these emissions including 1) building bake-out, 2) activated carbon filters to clean the air, 3) sealing or encapsulating the leveling material, and 4) removing the epoxy material and replacing it with an inorganic floor-leveling material.

Building bake-out was considered impractical because the TGA and GC/MS results indicated that an impractically high temperature (greater than 200 °C) would have to be applied for a long time to remove the phenol. In addition, previous studies have raised questions regarding the efficacy of building bake-out (11,12). Gaseous air cleaning using activated carbon filters was also considered as a mitigation approach. These filters would have to be periodically changed over the life of the building, since the phenol emissions would be expected to continue for such

a long time. It might also be possible to seal or encapsulate the leveling material by painting it, by covering it with a plastic sheet like polyethylene, or by a combination of both. If necessary, the effectiveness of this strategy could be enhanced by coupling it with activated carbon filters. The latter strategy, however, was judged unlikely to be cost-effective for several reasons. First, sealing or encapsulating the leveling material would cause phenol to be retained within the leveling material. Since the service lives of the sealing materials and plastic sheets would likely be much shorter than the life of the building, the air on Floors 2 through 5 would have to be periodically monitored for phenol over the life of the building to determine if and when resealing had become necessary. Moreover, since the life of the building is likely to be fifty years or more, there is a possibility that there would be a loss of institutional memory of the phenol emission problem.

The most certain, and probably the most cost-effective, long-term solution considered was to physically remove the leveling material and replace it with a light-weight portland cement-based material. This remedial strategy would eliminate the major source of phenol emissions. Replacement of the leveling material with a portland cement-based floor-leveling material would have the added advantage that any residual phenol left on the surface of the concrete slab coming in contact with the portland cement-based material would be converted into a salt, calcium phenolate. This should effectively prevent any significant quantity of residual phenol from being volatilized into the atmosphere.

## CONCLUSIONS

NIST performed a study to investigate whether phenol, or any other volatile organic compound, was being emitted by an epoxy floor-leveling material in an office building and, if so, to recommend remedial strategies. The chemical composition and physical properties of this material were studied using a variety of analytical procedures including thermogravimetric analysis, gas chromatography/mass spectrometry, liquid chromatography/mass spectrometry, and ultraviolet spectrophotometry. The following conclusions were derived from these analyses: 1) The epoxy floor-leveling material on Floors 2 through 5 contained significant concentrations of phenol and that phenol was emitted from the floor-leveling material at room temperature; also, phenol was the major organic emission from the material at room temperature; 2) The concentrations of phenol in the samples of epoxy floor-leveling material ranged from 0.25 to 0.52 % of the total mass of the leveling material; 3) The concrete floor slabs beneath the epoxy floor-leveling material were not contaminated by phenol; and, 4) The most certain, and probably the most cost-effective, long-term remedial strategy would be to physically remove the epoxy floor-leveling material and replace it with a light-weight portland cement-based product which contained no volatile organic compounds and would act as a scavenger for any traces of phenol.

This study is an example of the usefulness of a materials-science based approach to the diagnosis of indoor air quality problems. The physical understanding of the materials and material interactions involved, as well as the measurement procedures, were critical to arriving at the study's conclusions. Further progress in many areas related to indoor air quality are possible using similar approaches.

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